

# PATENT ABSTRACTS OF JAPAN

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) POSITIVE ELECTRODE ACTIVE MATERIAL FOR NON-AQUEOUS ELECTROLYTE SECONDARY BATTERY  
D ITS MANUFACTURING PROCESS

)Abstract:

OBLEM TO BE SOLVED: To provide a positive electrode active material, which can obtain a non-aqueous electrolyte secondary battery with which an initial electric discharge capacity as the secondary battery is maintained, and a electric charging/ discharging cycle characteristic under high temperature has been improved. LUTION: The positive electrode active material for non-aqueous electrolyte secondary batteries is composed t a titan oxide and/or lithium titanate is covered in a part of particle surface of lithium cobaltate particle rder, and the amount of the covering of the above titan oxide and/or lithium titanate is 2.0 to 4.0 mol % as Ti to alt in lithium cobaltate particle powder. The positive electrode active material can be obtained by that, after usting pH of the solution distributed with the lithium cobaltate particle, titanate is added, and then, making a ailed aqueous titanium oxide colloid stick to the particle surface of the lithium cobaltate particle, it is filtered, shed and dried to obtain the lithium cobaltate particle powder, to which the aqueous titanium oxide colloid is le to stick. Subsequently, the lithium cobaltate particle powder is heat-treated in oxidization atmosphere.

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AIMS

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im(s)]

im 1] Positive active material for nonaqueous electrolyte rechargeable batteries with which titanium oxide and/or  
um titanate are covered by a part of particle front face of cobalt acid lithium particle powder, and the amount of  
ering of said titanium oxide and/or lithium titanate is characterized by 2.0-4.0-mol being % as Ti to the cobalt in  
alt acid lithium particle powder.

im 2] The manufacturing method of the positive active material according to claim 1 for nonaqueous-electrolyte  
argeable batteries which obtains the cobalt acid lithium particle powder to which filtered, rinsed and dried and  
roxylation titanium colloid was made to stick after having adjusted pH of the water solution which distributed the  
alt acid lithium particle, adding the titanium salt subsequently and making detailed hydroxylation titanium colloid  
c to the particle front face of a cobalt acid lithium particle, and is subsequently characterized by to heat-treat this  
alt acid lithium particle powder in an oxidizing atmosphere.

im 3] The manufacturing method of the positive active material for nonaqueous electrolyte rechargeable batteries  
ording to claim 2 characterized by the temperature of heat treatment being 500 degrees C - 700 degrees C.

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## TAILED DESCRIPTION

ailed Description of the Invention]

01]

ustrial Application] This invention offers the positive active material which can obtain the nonaqueous electrolyte  
argeable battery with which the initial discharge capacity as a rechargeable battery was maintained, and the charge-  
-discharge cycle property under an elevated temperature has been improved.

02]

scription of the Prior Art] In recent years, portable-izing of electronic equipment, such as an AV equipment and a  
sonal computer, and cordless-ization are progressing quickly, and small and the demand to the rechargeable battery  
ch is lightweight and has a high energy consistency are high as these power sources for a drive. Charge and  
harge voltage is high under such a situation, and the rechargeable lithium-ion battery which has the advantage in  
ch charge-and-discharge capacity is also large attracts attention.

03] Conventionally, as positive active material useful to the rechargeable lithium-ion battery of a high energy mold  
a 4V class electrical potential difference, generally LiMn 2O4 of spinel type structure, LiMnO2 and LiCoO2 of  
c salt type structure, LiCo1-XNiXO2, and LiNiO2 grade are known, and especially, although excelled in the point of  
ing charge and discharge voltage with high LiCoO2, and charge-and-discharge capacity, the further property  
rovement is called for.

04] That is, since the equipment which operates with rechargeable batteries, such as a notebook computer, becomes  
elevated temperature with the use, to excel in the charge-and-discharge cycle property under an elevated temperature  
rechargeable battery is demanded. Moreover, although LiCoO2 can operate on a high electrical potential difference,  
action with the electrolytic solution tends to occur for the high voltage, and a charge-and-discharge cycle property  
is to fall.

05] Then, LiCoO2 excellent in the charge-and-discharge cycle property under an elevated temperature is demanded.

06] the approach (JP,4-329267,A --) of covering a cobalt acid lithium particle front face with a titanium compound  
ventionally for many property improvements of cobalt acid lithium particle powder JP,8-102332,A, JP,2000-  
605,A, etc., How (JP,6-44974,A etc.) to make titanium contain in a cobalt acid lithium particle, The approach (JP,7-  
124,A) using the mixture of cobalt acid lithium particle powder and a lithium titanium multiple oxide (LiTi 2O4) as  
itive active material is learned. Moreover, the approaches (the patent No. 3054829 official report, the patent No.  
4812 official report, the patent No. 2855877 official report, the patent No. 3003431 official report, etc.) of covering  
balt acid lithium particle front face with Lynn, boron, a zirconium dioxide, samarium oxide, etc. are learned.

07]

blem(s) to be Solved by the Invention] Although the positive active material which fulfills said many properties is  
ired most now and it is just going to be, it is not yet obtained.

08] namely, the above -- the effectiveness which controls a reaction with the electrolytic solution since the added  
nium atom is spread in the direction of the interior of a cobalt acid lithium particle and a titanium addition surface  
er is formed near the front face, although the approach of heat-treating is indicated by it after carrying out surface  
paration of the cobalt acid lithium particle front face by the titanium coupling agent to JP,4-329267,A -- it cannot  
ain -- the charge-and-discharge cycle property under an elevated temperature -- enough -- \*\*\*\* -- it is hard it saying  
reover, it is hard to say that it is excellent in industrial productivity since a coupling agent is expensive.

09] the above -- although carrying out distributed maintenance of the low activity oxide, such as a titanic-acid ghost,  
ndicated by JP,8-102332,A in a part of cobalt acid lithium particle front face, since the bonding strength of the  
nic-acid ghost currently held on the cobalt acid lithium particle front face is weak -- the charge-and-discharge cycle  
perty under an elevated temperature -- enough -- \*\*\*\* -- it is hard to say.

10] the above -- since the maldistribution part of the titanium compound particle to which mixing of a titanium compound particle becomes uneven and adheres occurs when a cobalt acid lithium particle and a titanium compound particle are blended dryly although the method of making a titanium particle and/or a titanium compound particle adhere to a cobalt acid lithium particle front face is indicated by JP,2000-200605,A -- the charge-and-discharge cycle property under an elevated temperature -- enough -- \*\*\*\* -- it is hard to say.

11] the above -- although the method of calcinating the mixture of lithium cobalt oxide and a titanate-acid ghost to JP,44974,A, and obtaining  $\text{Li}_{1.4}(\text{Co}_{0.7}\text{Ti}_{0.3})\text{O}_4$  is indicated, since the effectiveness which initial charge-and-discharge capacity falls, and controls a reaction with the electrolytic solution is not acquired -- the charge-and-discharge cycle property under an elevated temperature -- enough -- \*\*\*\* -- it is hard to say.

12] the above -- although the method of using the mixture of cobalt acid lithium particle powder and a lithium titanium multiple oxide ( $\text{LiTi}_2\text{O}_4$ ) as positive active material is indicated by JP,7-288124,A, since the effectiveness which controls a reaction with the electrolytic solution is not acquired only by making a lithium titanate-acid multiple oxide exist, it is hard to say that the charge-and-discharge cycle property under an elevated temperature improves.

13] moreover, the above -- since it is difficult to control a reaction with the electrolytic solution when it covers with different-species elements other than a titanium compound (Lynn, boron, a zirconium dioxide, samarium oxide, etc.) -- the charge-and-discharge cycle property under an elevated temperature -- enough -- \*\*\*\* -- it is hard to say.

14] Then, this invention makes it a technical problem to obtain the positive active material which was excellent in initial discharge capacity, and was excellent in the charge-and-discharge cycle property under an elevated temperature.

15] [Means for Solving the Problem] This invention as follows can attain said technical problem.

16] That is, titanium oxide and/or lithium titanate are covered by a part of particle front face of cobalt acid lithium particle powder, and this invention is positive active material for nonaqueous electrolyte rechargeable batteries with which the amount of covering of said titanium oxide and/or lithium titanate is characterized by 2.0-4.0-mol being % as to the cobalt in cobalt acid lithium particle powder.

17] Moreover, this invention adjusts pH of the water solution which distributed the cobalt acid lithium particle, and, subsequently adds a titanium salt. After making detailed hydroxylation titanium colloid stick to the particle front face of cobalt acid lithium particle, the cobalt acid lithium particle powder to which filtered, rinsed and dried and hydroxylation titanium colloid was made to stick is obtained. Subsequently It is the manufacturing method of said positive active material for nonaqueous electrolyte rechargeable batteries characterized by heat-treating this cobalt acid lithium particle powder in an oxidizing atmosphere.

18] Moreover, this invention is the manufacturing method of said positive active material for nonaqueous electrolyte rechargeable batteries characterized by the temperature of heat treatment being 500 degrees C - 700 degrees C.

19] It will be as follows if the configuration of this invention is explained in more detail.

20] First, the positive active material concerning this invention is described.

21] As for the positive active material concerning this invention, a part of particle front face of cobalt acid lithium particle powder is covered with titanium oxide and/or lithium titanate.

22] In this invention, when titanium oxide and/or lithium titanate have covered a part of particle front face of cobalt acid lithium particle powder and titanium oxide and/or lithium titanate cover the whole particle front face of cobalt acid lithium particle powder, initial discharge capacity falls. The content of titanium oxide and/or lithium titanate is 2.0-4.0-mol% to the cobalt of cobalt acid lithium particle powder in Ti conversion. In the case of not more than 2.0mol%, the effectiveness of the improvement in a cycle capacity maintenance factor is small, and in exceeding 4.0-mol%, initial charge capacity falls remarkably. desirable -- 2.1-3.5-mol% -- it is 2.2-3.0-mol% more preferably.

23] The mean particle diameter of the positive active material concerning this invention has desirable 1.0-10 micrometers. When mean particle diameter is less than 1.0 micrometers, since the fall of pack density and reactivity with the electrolytic solution increase, it is not desirable. Producing industrially becomes difficult in exceeding 10 micrometers.

24] The BET specific surface area of the positive active material concerning this invention has desirable 0.1-1.0  $\text{m}^2/\text{g}$ . Producing industrially becomes difficult when a BET specific surface area value is under 0.1  $\text{m}^2/\text{g}$ . Since the fall of pack density and reactivity with the electrolytic solution increase in exceeding 1.5  $\text{m}^2/\text{g}$ , it is not desirable.

25] As for the lattice constant of the positive active material concerning this invention, it is desirable that a-axis length is [ 2.81-2.82Å and c-axis length ] 14.045-14.065Å.

26] Next, the manufacturing method of the positive active material concerning this invention is described.

27] The positive active material concerning this invention adds alkali salt in the water solution which distributed the

alt acid lithium particle, subsequently adds a titanium salt, makes detailed hydroxylation titanium colloid stick to the front face of a cobalt acid lithium, obtains the cobalt acid lithium particle powder to which filtered, rinsed and dried and hydroxylation titanium colloid was made to stick, and, subsequently to the inside of an oxidizing atmosphere, obtained by heat-treating this cobalt acid lithium particle powder at 500-700 degrees C.

28] The cobalt acid lithium particle powder in this invention could be obtained by which approach of the solid phase unique which it is obtained by the usual approach, and mixes a lithium compound and a cobalt compound, for example, heat-treats, and is acquired, and the wet method which a lithium compound and a cobalt compound are made react in a solution, and obtains a cobalt acid lithium particle.

29] As for cobalt acid lithium particle powder, it is desirable that 0.1-1.5m<sup>2</sup>/g and a Li/Co ratio are [ mean particle diameter / 0.95-1.05, and a lattice constant ] 2.81-2.82Å of a-axis length and 14.045-14.065Å of c-axis length for 1.0-10 micrometers and a BET specific surface area value.

30] As alkali salt, a sodium hydroxide, a potassium hydroxide, a lithium hydroxide, etc. can be used. Especially, when a lithium hydroxide is used, it can consider as the mixture of lithium titanate or lithium titanate, and titanium oxide by being able to obtain the hydroxylation titanium colloid containing a lithium ion, and passing through heat treatment by adjusting an addition and a rinsing degree.

31] A titanium salt is added after adding alkali salt.

32] A titanium chloride, sulfuric-acid titanium, etc. can be used as a titanium salt.

33] As for the addition of a titanium salt, it is desirable that it is 2.0-4.0-mol% to the cobalt of cobalt acid lithium particle powder.

34] It is desirable by adding a titanium salt to set pH of a water solution to 10.0-12.0. It becomes difficult to make titanium-hydroxide colloid with pH of a water solution detailed when [ said ] out of range generate and adsorb.

35] As an ambient atmosphere of heat treatment, it is an oxidizing atmosphere and is among atmospheric air preferably. As heat treatment temperature, it is desirable that it is 500-700 degrees C. When a hydroxylation titanium rate remains in the case of less than 500 degrees C and it exceeds 700 degrees C, since sintering between particles occurs or a titanium atom is spread in the direction of the interior of a cobalt acid lithium particle, it is not desirable. 1 hours of the holding time are desirable. When shorter than 1 hour, a decomposition reaction is inadequate, and when longer than 5 hours, it is not desirable from the field of productivity and cost.

36] In manufacturing a positive electrode using the positive active material concerning this invention, according to a conventional method, it carries out addition mixing of an electric conduction agent and the binder. As an electric conduction agent, acetylene black, carbon black, a graphite, etc. are desirable, and polytetrafluoroethylene, polyvinylidene fluoride, etc. are desirable as a binder.

37] When manufacturing a rechargeable battery using the positive active material concerning this invention, it consists of said positive electrode, a negative electrode, and an electrolyte.

38] As a negative-electrode active material, a lithium metal, a lithium/aluminium alloy, a lithium / tin alloy, graphite, graphite, etc. can be used.

39] Moreover, as a solvent of the electrolytic solution, ethylene carbonate and the organic solvent which includes at least one kind of carbonate, such as propylene carbonate and dimethyl carbonate, and ether, such as dimethoxyethane, in addition to the combination of diethyl carbonate can be used.

40] Furthermore, as an electrolyte, at least one kind of lithium salt, such as 4 lithium perchlorate, lithium borate, etc. can be dissolved and used for the above-mentioned solvent in addition to a 6 phosphorus-fluoride acid lithium.

41] Initial discharge capacity is [ the capacity maintenance factor with a 140 - 150 mAh/g and a cycles / of 50 / of 60 degrees C after ] 97% or more, and the rechargeable battery manufactured using the positive active material concerning this invention has a low charge-and-discharge capacity in a overcharge trial.

42]

[ embodiment of the Invention] The gestalt of typical operation of this invention is as follows.

43] About identification of the product after surface treatment and baking, the powder X diffraction (alpha 40kV [ of 15kV Cu-K ] 40mA) was used. Moreover, the lattice constant was calculated from each diffraction peak of said powder X diffraction.

44] Moreover, plasma-emission-spectrometry equipment ( SPS4000 made from the SEIKO electronic industry) was used for elemental analysis.

45] The cell property of positive active material prepared a positive electrode, a negative electrode, and the electrolytic solution, and produced and evaluated the cell cell of a coin mold by the following manufacturing method.

46] after it weighs precisely the polyvinylidene fluoride of the acetylene black which is <production of positive electrode> positive active material, and an electric conduction agent, and a binder so that it may be set to 85:10:5 by the

ght ratio, and fully mixing with a mortar, it distributes to a N-methyl-2-pyrrolidone -- making -- a positive electrode mixture -- the slurry was adjusted. Next, after applying this slurry to the aluminium foil of a charge collector by micrometer thickness and carrying out the vacuum drying at 150 degrees C, it pierced to disc-like [ phi 16mm ], and sidered as the positive-electrode plate.

[7] The <production of negative electrode> metal lithium foil was pierced to disc-like [ phi 16mm ], and the negative trode was produced.

[8] One mol (LiPF<sub>6</sub>) /of 6 phosphorus-fluoride acid lithiums was mixed l. as an electrolyte to the mixed solution of volume ratio 50:50 of <preparation of the electrolytic solution> ethylene carbonate, and diethyl carbonate, and it sidered as the electrolytic solution.

[9] The electrolytic solution was further poured in through the separator made from polypropylene using the case e from SUS316 between the above-mentioned positive electrode and the negative electrode all over the glove partment of a <assembly of coin mold cell cel> argon ambient atmosphere, and the coin cell of CR2032 mold was luced.

[0] <Cell evaluation> The charge and discharge test of a rechargeable battery was performed using said coin mold As a Measuring condition, current density to a positive electrode was made into 0.2 mA/cm<sup>2</sup> under the temperature 0 degrees C, and cut-off voltage repeated charge and discharge between 3.0V and 4.25V. Moreover, about the charge trial, it charged to 4.95V under the temperature of 20 degrees C.

[1] The lithium carbonate and cobalt oxide of the specified quantity were fully mixed so that the mole ratio of a manufacture of positive active material> lithium and cobalt might be set to 1:1, under the oxidizing atmosphere, it inated at 900 degrees C for 10 hours, and cobalt acid lithium particle powder was obtained.

[2] For the diameter of an average major axis, 8.0 micrometers and a BET specific surface area value were [ 0.6m<sup>2</sup>/g the lattice constant of the obtained cobalt acid lithium particle powder ] 2.817A of a-axes, and 14.057A of c-axes.

[3] Next, the obtained cobalt acid lithium particle was distributed in the water solution, and the lithium hydroxide thrown in. Next, the titanium tetrachloride which contains 2.5-mol % of titanium to cobalt was supplied, pH of a tion was adjusted to the 12 neighborhoods, and the cobalt acid lithium with which the detailed hydroxylation ium colloid containing a lithium ion stuck to the particle front face by passing through rinsing and a desiccation ess was obtained. Subsequently, positive active material was obtained by calcinating the cobalt acid lithium particle hich the obtained hydroxylation titanium colloid stuck at 500 degrees C under an oxidizing atmosphere for 5 hours.

[4] For 8.0 micrometers and a BET specific surface area value, the diameter of an average major axis was [ the ce constant of the obtained positive active material / the content of 2.816A of a-axes, 14.049A of c-axes, and ium ] 2.20-mol% to cobalt to 0.5m<sup>2</sup>/g. What, as for a titanium content, the whole quantity remains mostly to an tion, and exists in the state of lithium titanate on a particle front face, without doping titanium in the grid of a cobalt lithium since the lattice constant after baking does not change as compared with covering processing before can be umed. Moreover, since the obtained positive active material is the same as that of the diffraction pattern of the X action of the cobalt acid lithium particle powder before processing as shown in drawing 1 and 2, titanium oxide /or lithium titanate can be presumed to be what is covered by the front face of a cobalt acid lithium particle, without ting in single phase.

[5] For initial discharge capacity, the capacity maintenance factors with a 150 mAh/g and a cycles [ of 50 ] of 60 rees C after were [ 97% / 50cycle and the overcharge trial of the coin mold cell produced using said positive active erial ] 250 mAh/g.

[6] action] The positive active material which the most important point requires for this invention in this invention is the it of excelling in the charge-and-discharge cycle property under an elevated temperature, holding the initial harge capacity as a rechargeable battery by covering a part of cobalt acid lithium particle front face with titanium le and/or lithium titanate and.

[7] The detailed titanium oxide particle and/or the lithium titanate particle are chemically combined with a part of icle front face of a cobalt acid lithium particle by setting to this invention, making hydroxylation titanium colloid ctly detailed on a cobalt acid lithium particle front face generate and adsorb, and subsequently heat-treating in an lizing atmosphere by the wet reaction.

[8] Therefore, mixing is not uneven, or since a mutual particle is only mere physical adsorption, the effectiveness of invention is not acquired at the case which blended dryly the cobalt acid lithium particle, the titanium oxide particle, he lithium titanate particle. Moreover, also when it heat-treats after mixing with hydroxylation titanium or lithium ate, since it will not be in the uniform mixed state, the effectiveness of this invention is not acquired.

[9] It is based on having made titanium oxide and/or lithium titanate contain in the range in which the initial

large capacity which an original cobalt acid lithium particle has is not reduced that initial discharge capacity can be maintained in this invention.

50] In this invention, an elevated-temperature property is improvable by using the positive active material which a front face of a cobalt acid lithium particle covered with titanium oxide and/or lithium titanate, because of the particle surface section and the reaction (oxidative degradation) of the electrolytic solution which are effected with the time of an elevated temperature (60 degrees C) or the high potential beyond 4.8V are controlled.

51] Next, the example of a comparison is given in an example list.

52] Except having changed various additions of examples 1-3, the example 1 of a comparison - 4 titanium salt, and treatment conditions, positive active material was manufactured like the gestalt of implementation of said invention, and, subsequently the coin mold cell was manufactured.

53] Many properties of the positive active material obtained in Table 1 in the manufacture conditions at this time and cell property of a coin mold cell are shown in Table 2.

54] In addition, surface treatment was not performed in the example 1 of a comparison. It did not heat-treat in the examples 2 and 3 of a comparison. The example 4 of a comparison performed heat treatment conditions at 900 degrees

55]  
Table 1]

	製造法					
	アルカリ塩	Ti化合物の種類	処理量 (mol%)	表面処理物	熱処理温度 (°C)	熱処理時間 (hr)
例 1	水酸化リチウム	四塩化チタン	2.5	TiO <sub>2</sub> ·2H <sub>2</sub> O	700	1
例 2	水酸化ナトリウム	四塩化チタン	2.5	TiO <sub>2</sub> ·2H <sub>2</sub> O	500	5
例 3	水酸化リチウム	硫酸チタン	3.0	TiO <sub>2</sub> ·2H <sub>2</sub> O	500	5
例 4	水酸化リチウム	硫酸チタン	3.0	TiO <sub>2</sub> ·2H <sub>2</sub> O	700	1
例 1	---	---	0	なし	熱処理なし	
例 2	水酸化リチウム	四塩化チタン	2.5	TiO <sub>2</sub> ·2H <sub>2</sub> O	熱処理なし	
例 3	水酸化リチウム	四塩化チタン	3.0	TiO <sub>2</sub> ·2H <sub>2</sub> O	熱処理なし	
例 4	水酸化リチウム	四塩化チタン	2.5	TiO <sub>2</sub> ·2H <sub>2</sub> O	900	1

66]  
Table 2]

	粉体特性					電池特性		
	平均 粒子径 μm	BET 比表面積 m <sup>2</sup> /g	格子定数(Å)		Ti含有量 (mol%)	1stdis. (mAh/g)	C.E.60°C (%/50cycle)	過充電 容量 20°C (mAh/g)
			(a)	(c)				
例 1	8.0	0.3	2.816	14.049	2.17	148	99	256
例 2	8.0	0.5	2.817	14.053	2.22	150	98	252
例 3	8.0	0.6	2.816	14.049	2.79	141	97	242
例 4	8.0	0.3	2.816	14.049	2.71	144	97	245
例 1	8.0	0.3	2.817	14.053	0	150	90	270
例 2	8.0	6.9	2.817	14.053	2.25	125	92	240
例 3	8.0	8.2	2.817	14.053	2.76	118	88	225
例 4	8.0	0.3	2.805	14.001	2.20	135	90	250

67] Initial discharge capacity holds 140 or more mAh/g, and the cell property of the coin cell produced using the positive active material concerning this invention has a capacity maintenance factor with a cycles [ of 50 ] of 60 degrees after in 97% or more and high level. Furthermore, also in a overcharge trial, the value is decreasing as compared with charge capacity before covering processing, and reaction control with the particle front face of positive active

material and the electrolytic solution is suggested.

68] Moreover, only by covering hydroxylation titanium, although a reduction extreme in overcharge capacity is checked, initial discharge capacity is also as low as near 125 mAh/g to coincidence, and an improvement effect is not obtained about a cycle capacity maintenance factor, either, as shown in the example of a comparison.

69]

Effect of the Invention] The nonaqueous electrolyte rechargeable battery with which the initial discharge capacity as a rechargeable battery was maintained, and the elevated-temperature property has been improved by using the positive electrode material concerning this invention can be obtained.

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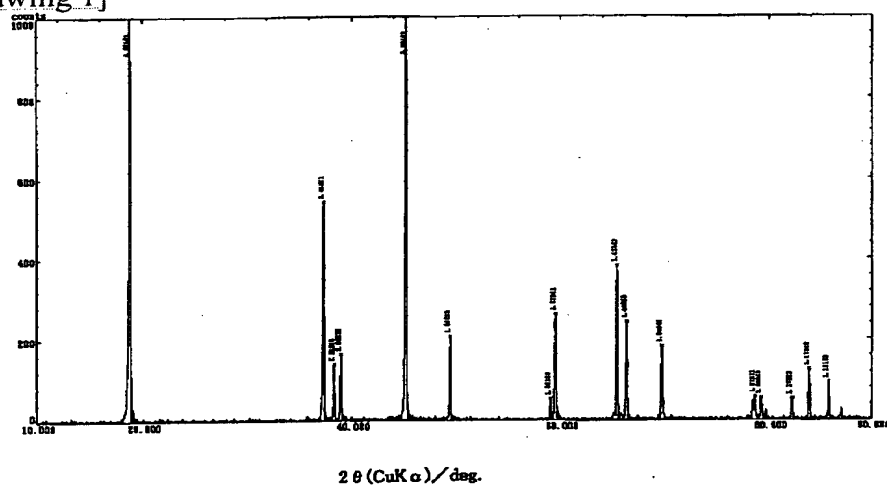
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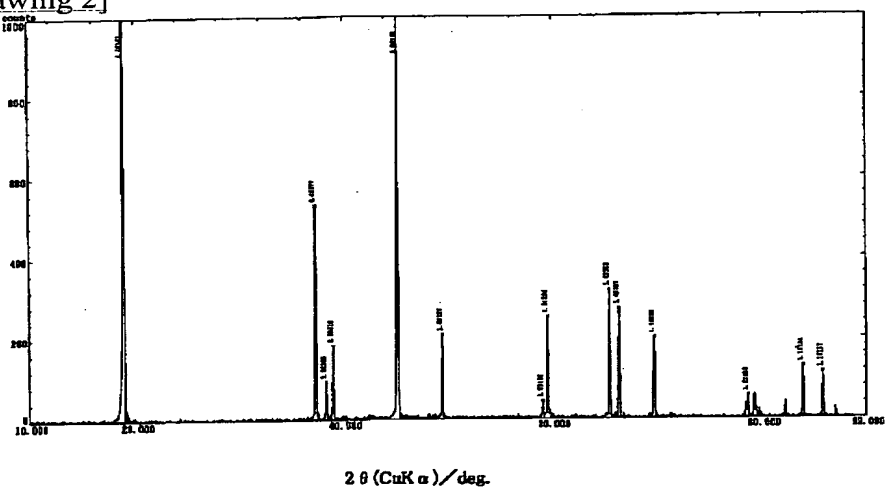
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## AWINGS

awing 1]



awing 2]



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